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# मानक

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IS 8861 (1978): 1, 4-dichloro-2-nitrobenzene [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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IS : 8861 - 1978

*Indian Standard*  
SPECIFICATION FOR  
1, 4-DICHLORO-2-NITROBENZENE

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**INDIAN STANDARDS INSTITUTION**  
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# Indian Standard

## SPECIFICATION FOR

### 1, 4-DICHLORO-2-NITROBENZENE

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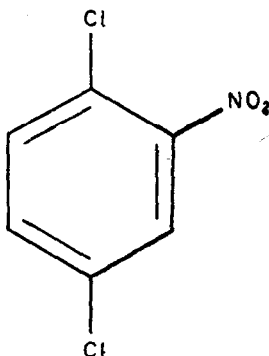
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*Indian Standard*  
SPECIFICATION FOR  
1, 4-DICHLORO-2-NITROBENZENE

0. FOREWORD

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 8 June 1978, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

**0.2** 1, 4-Dichloro-2-nitrobenzene ( $C_6H_3O_2NCl_2$ ) is an important dye intermediate used in the manufacture of dyes. It is represented by the following structural formula:



1, 4-Dichloro-2-Nitrobenzene ( Molecular Mass 192 )

**0.3** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960\*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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\*Rules for rounding off numerical values (*revised*).

## 1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for 1, 4-dichloro-2-nitrobenzene.

## 2. REQUIREMENTS

2.1 **Description** — The material shall be in the form of brownish crystalline solid.

2.2 The material shall also comply with the requirements given in Table 1.

**TABLE 1 REQUIREMENTS FOR 1, 4-DICHLORO-2-NITROBENZENE**  
( *Clauses 2.2, 4.3.1, 4.3.2 and 5.1* )

SL No.	CHARACTERISTIC	REQUIREMENT ( ON DRY BASIS )	METHOD OF TEST, REF TO CL NO. IN APPENDIX A
(1)	(2)	(3)	(4)
i)	Crystallizing point, °C, <i>Min</i>	53.0	A-2
ii)	Assay, percent by mass, <i>Min</i>	99.5	A-3
iii)	3, 4-Dichloronitrobenzene, percent by mass, <i>Max</i>	0.1	A-4

## 3. PACKING AND MARKING

3.1 **Packing** — The material shall be packed in steel drums ( *see* IS : 2552-1970\* ) or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

3.2 **Marking** — Each container shall bear legibly and indelibly the following information:

- Name of the material;
- Name of the manufacturer and his recognized trade-mark, if any;
- Tare, net and gross mass;
- Batch number; and
- The minimum cautionary notice worded as under:

‘ TOXIC ! HANDLE WITH CARE ! ’

\*Specification for steel drums ( galvanized and ungalvanized ) ( *first revision* ).



**3.2.1** The containers may also be marked with the ISI Certification Mark.

**NOTE** — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution ( Certification Marks ) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

## 4. SAMPLING

**4.1** Representative samples of the material shall be drawn as prescribed in 3 of IS : 5299-1969\*.

### 4.2 Number of Tests

**4.2.1** Tests for crystallizing point and assay shall be conducted on each of the individual samples.

**4.2.2** Tests for 3, 4-dichloronitrobenzene shall be conducted on the composite sample.

### 4.3 Criteria for Conformity

**4.3.1** *For Individual Samples* — The lot shall be declared as conforming to the requirements of crystallizing point and assay if each of the individual test results satisfies the relevant requirements given in Table 1.

**4.3.2** *For Composite Sample* — For declaring the conformity of the lot to the requirement of 3, 4-dichloronitrobenzene tested on the composite sample ( *see* 4.2.2 ) the test result for this characteristic shall satisfy the relevant requirement given in Table 1.

**4.3.3** The lot shall be declared to conform to the requirements of the specification if conditions laid down in 4.3.1 and 4.3.2 are satisfied.

## 5. TEST METHODS

**5.1** Tests shall be carried out according to the methods prescribed in Appendix A, as indicated in col 4 of Table 1.

**5.2 Quality of Reagents** — Unless specified otherwise, pure chemicals and distilled water ( *see* IS : 1070-1977† ) shall be employed in tests.

**NOTE** — ' Pure Chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

\*Methods of sampling and tests for dye intermediates.

†Specification for water for general laboratory use ( *second revision* ).

## APPENDIX A

(Table 1, and Clause 5.1)

### METHODS OF TEST FOR 1, 4-DICHLORO-2-NITROBENZENE

#### A-1. PREPARED SAMPLE

**A-1.1** Dry the material at  $45^{\circ} \pm 1^{\circ}\text{C}$  under vacuum. Grind and mix well. Transfer the material to a wide mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this *prepared sample* for tests.

#### A-2. DETERMINATION OF CRYSTALLIZING POINT

**A-2.1** Determine the crystallizing point of the material as prescribed in 7 of IS : 5299-1969\*. Use *prepared sample* ( see **A-1.1** ) for the test.

#### A-3. ASSAY

**A-3.1** Assay the material according to the method prescribed in 14 of IS : 5299-1969\*. Use *prepared sample* ( see **A-1.1** ) for the test.

#### A-4. DETERMINATION OF 3, 4-DICHLORONITROBENZENE

**A-4.0 Outline of the Method** — The isomer 3, 4-dichloronitrobenzene in 1, 4-dichloro-2-nitrobenzene is determined by thin-layer chromatography after reducing the nitro compound to the amino compound.

##### A-4.1 Apparatus

**A-4.1.1 Thin Layer Glass Plate** — of size  $10 \times 20$  cm coated uniformly with silica gel-G or an equivalent powder in a thickness of 250-micron and activated at  $110^{\circ}\text{C}$  for 1 hour.

**A-4.1.2 Micropipette**

**A-4.1.3 Developing Chamber**

**A-4.1.4 Chromatographic Sprayer**

##### A-4.2 Reagents

**A-4.2.1 1, 4-Dichloro-2-Nitrobenzene** — pure, free from isomers.

**A-4.2.2 3, 4-Dichloronitrobenzene** — pure.

**A-4.2.3 Glacial Acetic Acid**

**A-4.2.4 Zinc Dust**

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\*Methods of sampling and tests for dye intermediates.

**A-4.2.5 Benzene****A-4.2.6 Sodium Nitrite****A-4.2.7 Hydrochloric Acid**

**A-4.2.8 Spray Reagent** — Dissolve 1 g of 1N naphthylethylene diamine dihydrochloride in a mixture of 50 ml of methanol and 50 ml of 10 percent hydrochloric acid ( *v/v* ).

**A-4.2.9 Sodium Hydroxide Solution** — 20 percent ( *m/v* ).

**A-4.3 Procedure**

**A-4.3.1** Weigh 1 g each of the pure 1, 4-dichloro-2-nitrobenzene into 4 beakers of 500-ml capacity each. Weigh 0.1 g of 3, 4-dichloronitrobenzene into a dry 100-ml volumetric flask, dissolve and make up to volume with glacial acetic acid. Pipette out (using a safety pipette) 0.5 ml, 1 ml, 2 ml and 3 ml of this acetic acid solution into the 4 beakers containing pure 1, 4-dichloro-2-nitrobenzene. Thus we have standards containing 0.05 percent, 0.1 percent, 0.2 percent and 0.3 percent of 3, 4-dichloronitrobenzene. Now add 20 ml of glacial acetic acid to each beaker to dissolve the contents, warm, if necessary. Add 5 g of zinc dust to each beaker, followed by 10 ml of hydrochloric acid. Keep for 30 minutes. Again add 5 g more of zinc dust and 10 ml of hydrochloric acid and keep for 30 minutes. Repeat the operation for a third time. Now make the contents of all the beakers alkaline using sodium hydroxide solution. Add 25 ml of benzene and stir well to extract the amine formed. Transfer the benzene layer to a separating funnel by decantation. Repeat the extraction with two more instalments of 25 ml each of benzene and collect all the benzene extracts in the same separating funnel. Separate the benzene layer and transfer it into a 100-ml volumetric flask.

**A-4.3.2** Prepare the amine extract of the material under test in the same way as in the case of the four standards ( *see A-4.3.1* ).

**A-4.3.3** Spot 10 microlitre each of the sample solution and the four standards on the thin layer plate. Allow to dry and keep the plate in the developing chamber containing benzene solvent. Allow to run for about 30 minutes. Take out the plate and allow to dry. Keep the plate for 2 minutes in a chamber containing nitrous acid fumes ( prepared by mixing sodium nitrite and hydrochloric acid ). Blow out the excess nitrous fumes from the plate and spray with the spraying reagent. The sample and the impurities are seen as violet spots. The material has *R<sub>f</sub>* value of 0.95 and the 3, 4-dichloronitrobenzene 0.69. Compare the intensity of the impurity in the sample to the nearest in the standards.

# INTERNATIONAL SYSTEM OF UNITS ( SI UNITS )

## Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

## Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

## Derived Units

QUANTITY	UNIT	SYMBOL	CONVERSION
Force	newton	N	1 N = 0.101 972 kgf
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m <sup>2</sup>
Frequency	hertz	Hz	1 Hz = 1 c/s (s <sup>-1</sup> )
Electric conductance	siemens	S	1 S = 1 A/V
Pressure, stress	pascal	Pa	1 Pa = 1 N/m <sup>2</sup>